

Assessment of Variations in Thermal Cycle Life Date of Thermal Barrier Coated Rods

Robert C. Hendricks and G. McDonald
Lewis Research Center
Cleveland, Ohio

Prepared for the
International Conference on Metallurgical Coatings
sponsored by the American Vacuum Society
San Francisco, California, April 6-10, 1981



ASSESSMENT OF VARIATIONS IN THERMAL CYCLE LIFE DATA
OF THERMAL BARRIER COATED RODS

R. C. Hendricks and G. McDonald
National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

ABSTRACT

An analysis of thermal cycle life data for 22 thermal barrier coated (TBC) specimens is presented. The $\text{ZrO}_2\text{-}8\text{Y}_2\text{O}_3/\text{NiCrAlY}$ plasma spray coated Rene 41 rods were tested in a Mach 0.3-Jet A/air burner flame.

All specimens were subjected to the same coating and subsequent test procedures in an effort to control three parametric groups; material properties, geometry and heat flux.

Statistically, the data sample space had a mean of 1330 cycles with a standard deviation of 520 cycles. The data were described by normal or log-normal distributions, but other models could also apply; the sample size must be increased to clearly delineate a statistical failure model.

The statistical methods were also applied to adhesive/cohesive strength data for 20 TBC discs of the same composition, with similar results. The sample space had a mean of 9 MPa (1.3 ksi) with a standard deviation of 4.2 MPa (0.6 ksi).

INTRODUCTION

Thermal stresses experienced by thermal barrier coatings (TBC's) are controlled primarily by temperature level and rate of temperature change to attain that level. Other factors such as salt deposition, coating microstructural changes, diffusion and oxidation effects are also consequences of time at temperature and rate to attain that temperature.

In reference 1, the life cycle effects of stresses at a cylindrical interface between the $\text{ZrO}_2\text{-}12\text{ wt}\% \text{Y}_2\text{O}_3$ and NiCrAlY were found to be a

function of rate of heating. Although it was clear that multiple short cycling was detrimental to total specimen lifetime, the variation in failure data was quite large. As the calculated radial stress was within the range of adhesive/cohesive strength data given in reference 2, it was assumed that the failures were due to excessive stress. To further illustrate the possible effects of heating rate, flat specimens at high (F/A) ran <370 cycles and at low (F/A) specimens ran >4700 cycles, a very large difference in cyclic life, reference 3.

In the tests of references 1 to 3, our general experience with rods, tubes and flat plate specimens, and that of others such as reference 4, indicate there are large variations in coating parameters such as cyclic life or adhesive/cohesive strength (possibly of the same order as the sample mean).

The purpose of this paper is to examine: (1) variations in cyclic life, (2) variations in adhesive/cohesive coating strength, and (3) some possible influences such as heating rate, stress reversal, temperature level and ceramic deposition methods on coating life.

Dr. Steven Sidik provided statistical codes and theoretical assistance for this investigation.

EXPERIMENTAL PROCEDURE

Materials and Coatings

Twenty-two solid Rene 41 rods 13 mm in diameter were coated with a NiCrAlY bond coat and $\text{ZrO}_2\text{-Y}_2\text{O}_3$ ceramic by first grit blasting with Al_2O_3 and then spraying in air with an Ni-18Cr-12Al-0.3Y bond coat, nominally 0.13 mm thick. The specimens were then coated with a nominal 0.38 mm layer of zirconia pre-alloyed with 8 wt% Y_2O_3 .

To measure adhesive/cohesive strength, twenty solid flat piston heads (discs) 25 mm in diameter were coated using these same procedures and coating thicknesses.

All specimens were subjected to the same procedure as a control on the parametric groups of material properties and geometry.

Apparatus and Procedure

The 22 coated rods were evaluated by heating eight specimens at a time in a rotating carousel with a 0.3 Mach burner rig firing Jet A-1. The free stream gas temperature was approximately 1450° C (2460° F). The specimens were heated for 5 min. followed by 3 min. of cooling; the average specimen temperature cycled between ambient and 1038° C (1900° F). The condition of the ceramic coating was determined by visual inspection; when coating failure occurred, a newly coated rod replaced it. In this manner, an attempt was made to control the parametric group for heat flux (heating rate and level).

The burner was moved by a pneumatic cylinder to impinge on the specimens in less than 1 sec. Measurements of the rise in temperature after impingement of the flame are reported in references 1 and 3.

The 20 coated "piston heads" were epoxied to mating pistons in a cylindrical guide. After curing, the assembly was removed, placed in a testing machine with gimble and swivel axes for alignment flexibility and pulled until coating failure occurred. The procedure, similar to that of reference 2, provides adhesive/cohesive strength data for the coating material.

RESULTS AND DISCUSSION

The results of cyclic heating of a total of 22 ceramic coated rods in the 0.3-Mach flame are shown in figure 1. The cycles to failure for the

ceramic coatings are plotted against the respective cumulative distribution function $F(x)$.

These data appeared to follow several failure distributions, normal, Weibull, log-normal, gamma (or chi-square), as shown in figures 1(a) to (d), respectively.*

The average life is 1330 cycles with a standard deviation of 520 cycles which is quite large.

It should be noted that the variations in life cycle data are as much as a factor of five; this is larger than anticipated for other failure mechanisms, e.g., creep of materials (ref. 9), and perhaps larger than for low cycle fatigue.

Removal of one of the first three early failure points, figures 1(a) and (b), reveals a distinct curvature to the data locii. Disregarding at least two of these same three points, the locus of points on figure 1(c) is linear, implying that our life cycle data, beyond early failures, are log-normally distributed. These data appear to follow two distributions, normal at early times and log-normal at later times implying the hazard function distribution may not be monotone.

An analysis of the gamma (or chi-square) distributions also leads to a reasonable representation of the data, figure 1(d). The gamma distribution is an attempt to represent the reproducible thermal loads applied to the specimens each cycle.

In essence, early failures occur perhaps more often than one would expect, long term failures occur less often than one would expect and the standard deviation is much larger than desired. They may stem from a single

*Ignoring the "tails," many distributions are similar and tend to appear normal.

cause such as interface failure. It may be that the lower life cycle times, figure 1, are due to ZrO_2 failures as a result of abnormal spraying. Examination of the early failures revealed little evidence to explain why they failed and at this time early failures remain unexplained.

In an effort to further understand the significance of these data and perhaps postulate a model, a more detailed analysis of the normal and log-normal distributions was carried out using the program MINITAB, reference 5, adapted from OMNITAB, reference 6, and the anticipated sample behavioral results of references 7 and 8 for normally distributed sample spaces of limited size. Examination of the correlation coefficients for subsets of the sample space obtained by dropping early and late failures gave no significant discrimination between the normal and log-normal distributions. While a marginally better statistical correlation is found for the normal distribution, verification or denial of whether the model should be normal or log-normal (or perhaps gamma or Weibull) must await a much larger sample size. The data did not appear to follow the extreme value failure distribution.

The radial thermal stresses produced at the interface were related to the adhesion/cohesive test data, references 1 and 2. The results of the 20 ceramic coated piston adhesive/cohesive strength tests are shown in figure 2.

The average adhesive/cohesive pulloff stress is 9 MPa (1.3 ksi) with a standard deviation of 4.2 MPa (0.61 ksi).

Applying the same statistical approach as described previously for the 22 coated rods, these data also appear to follow several failure distributions including the early failures.

The correlation coefficients for the 20 member experimental sample space, 0.966 and 0.980 for the normal and log-normal distributions, respec-

tively, was greater than anticipated (0.95) for 90-percent of the sample space for a theoretical normally distributed sample of 20 members.

The reported data, apparently limited to reference 2, is for four ZrO_2 -12 wt% Y_2O_3 coated "pistons." Applying the same statistical approach as for the previous sample spaces, the average pulloff stress was found to be 6.1 MPa (0.89 ksi) with a standard deviation of 1.9 MPa (0.27 ksi) and a correlation coefficient of 0.92. Applying a t-test to the 4 specimen and the 20 specimen sample spaces, there exists a significant overlap of the mean values. This implies that there may not be a significant difference between the sample spaces. Further test data are required.

The 22-TBC Mach 0.3 rod specimen data set, the 20-piston adhesion/cohesion pulloff specimen set and our general experience indicate a wide variation in coating failures with and without heating. We now want to explore four possible failure mechanisms which could contribute to the large variation in the experimental data.

Heating rate effect: in the initial part of the heating cycle, the ZrO_2 -NiCrAlY radial interface stress becomes large and tensile while stresses parallel to the plane are compressive. The magnitude depends on the rate of heating. The sensitivity of these stresses to heating rate, reference 1, coupled with the significant variation in the range of the adhesion/cohesion data, reference 2 and those herein, implies that variations in heating rate can produce significant variations in the life cycle data.

Stress Reversal: in the intermediate part of the heating cycle the radial stresses change from tensile to compressive. The interface is thus subjected to a complete stress reversal each thermal cycle which implies cyclic fatigue at the interface.

Strain Relief: near 955°C (1750°F), the allowable stress of the NiCrAlY becomes less than for ZrO_2 . Since the interface strength may be due to a combined mechanical "chemical" interlocking bond, the implication is that the base can no longer properly restrain the ZrO_2 thereby possibly degrading the bond via strain relief. The latter would be quite difficult to demonstrate as the deterioration would not be noted until failure occurs.

Ceramic Void: the strength of the ZrO_2 is substantially greater than the adhesive/cohesive strength at the interface and generally the failures occur at or near the interface. Sometimes they occur in the ZrO_2 perhaps due to the formation of a void during spraying. Abnormal spraying can be caused by a maldistribution of the $\text{ZrO}_2\text{-Y}_2\text{O}_3$ either being fed into the plasma gun or within the plasma proper, variation in stand-off distance and/or application pattern, thickness, etc. Here we find a very hot jet being focused onto the coating surface perhaps in violation of the heating rate criteria. We feel that a complete thermographic study of the plasma spraying process is warranted.

SUMMARY

Life cycle data for 22 thermal barrier coated rods ($\text{ZrO}_2\text{8Y}_2\text{O}_3/\text{Ni-18Cr-12Al-0.3Y}$ on Rene 41) subjected to cyclic exposure to a Mach 0.3 burner flame (ambient to 1038°C (1900°F)) were tested and found to be statistically modeled by normal or log-normal distributions. Other statistical models, such as gamma or Weibull, could also apply to this limited sample space. The sample mean was 1330 cycles with a standard deviation of 520 cycles. Failures occur primarily at the interface between the ZrO_2 and the NiCrAlY layers.

Adhesive/cohesive pulloff strength data for 20 thermal barrier coated flat head piston specimens were taken. The coatings and plasma spraying

procedures were the same as for the 22-TBC rods and the test data were analyzed by the same statistical methods. The average pulloff stress was 9 MPa (1.3 ksi) with a standard deviation of 4.2 MPa (0.61 ksi). The t-test on these TBC data 20 (ZrO_2 -8 wt% Y_2O_3) and four (ZrO_2 -12% Y_2O_3) specimens do not clearly demonstrate significant differences. It is most important that the test sample size be substantially increased in an effort to more clearly define statistical failure.

Calculated interface stresses, from previous work, indicate that the radial stress is of about the same magnitude as the adhesive/cohesive strength. Failures are perhaps due to the combined effects of excessive heating rate, thermal fatigue, and relaxation of the ZrO_2 and or the interface bond; these elements are heavily dependent on temperature level and the rate to attain that level. Some of the lower life cycle data may be due to ZrO_2 failures. At longer total time, oxidation will become a factor.

REFERENCES

1. G. McDonald and R. C. Hendricks, NASA Tech. Memo. TM-81480, 1980.
2. S. R. Levine, NASA Tech. Memo. TM-73792, 1978.
3. R. C. Hendricks, G. McDonald, and N. J. Poolos, Int. Coatings Conf., American Vac. Soc., San Francisco, 1981.
4. C. A. Anderson, R. J. Bratton, S. K. Lee, J. Allen, K. E. Munsen, K. L. Rieke, 5th Quarterly Progress Report to NASA DEN 3-110, Westinghouse, 1981.
5. T. A. Ryan, B. L. Joiner, F. Ryan, MINITAB Student Handbook. Duxbury Press, North Scituate, MA, 1976.
6. J. Hilsenrath, OMNITAB; A Computer Program for Statistical and Numerical Analysis, Handbook 101, National Bureau of Standards, Washington, DC, 1968.

7. C. Daniel, Applications of Statistics to Industrial Experimentation, Wiley, New York, 1976.
8. T. A. Ryan and B. L. Joiner, Normal probability plots and tests for normality. Penn. State Univ. Dept. of Statistics.
9. O. J. Horger (ed.), ASME Handbook. Vol. 1. Metals Engineering Design, 2nd ed., McGraw-Hill, New York, 1965.
10. R. E. Barlow, F. Proschan, L. C. Hunter, Mathematical Theory of Reliability, the SIAM Series in Applied Mathematics, Wiley, New York, 1965.

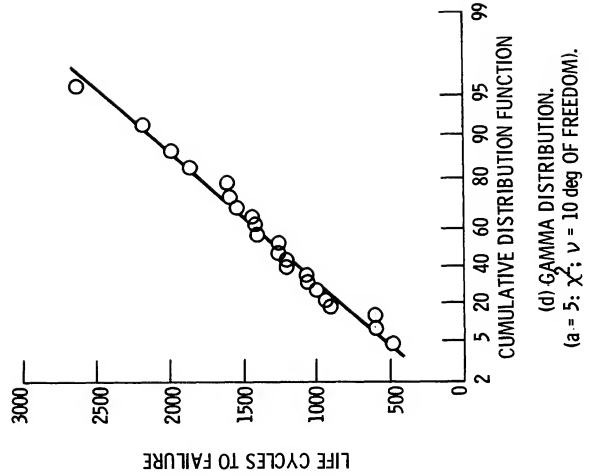
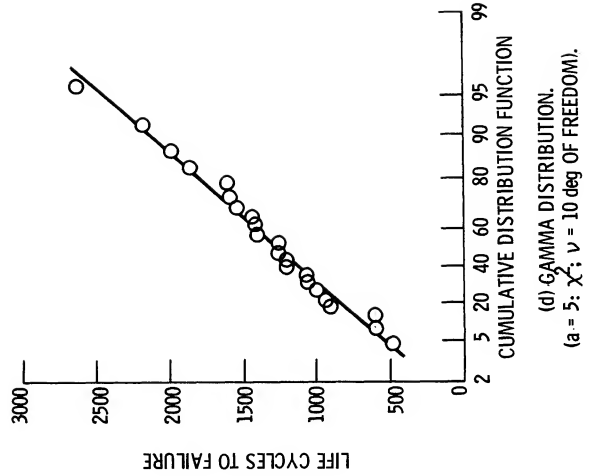
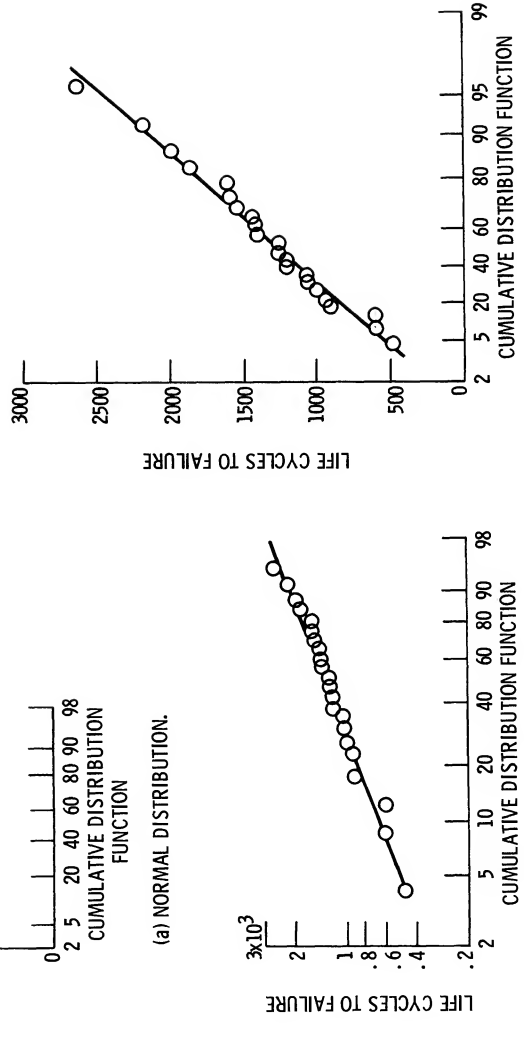
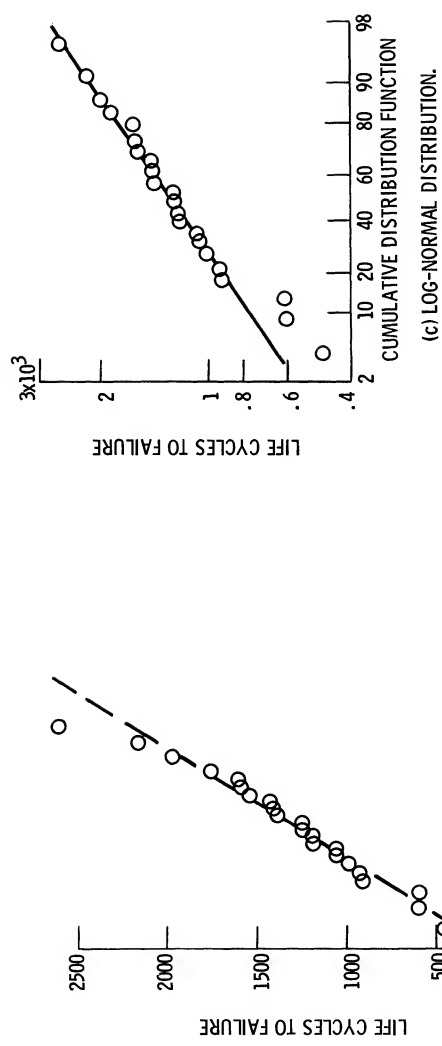


Figure 1. - Life cycle failure of 22-13 mm diameter thermal barrier coated rod specimens in a 0.3 Mach burner flame.

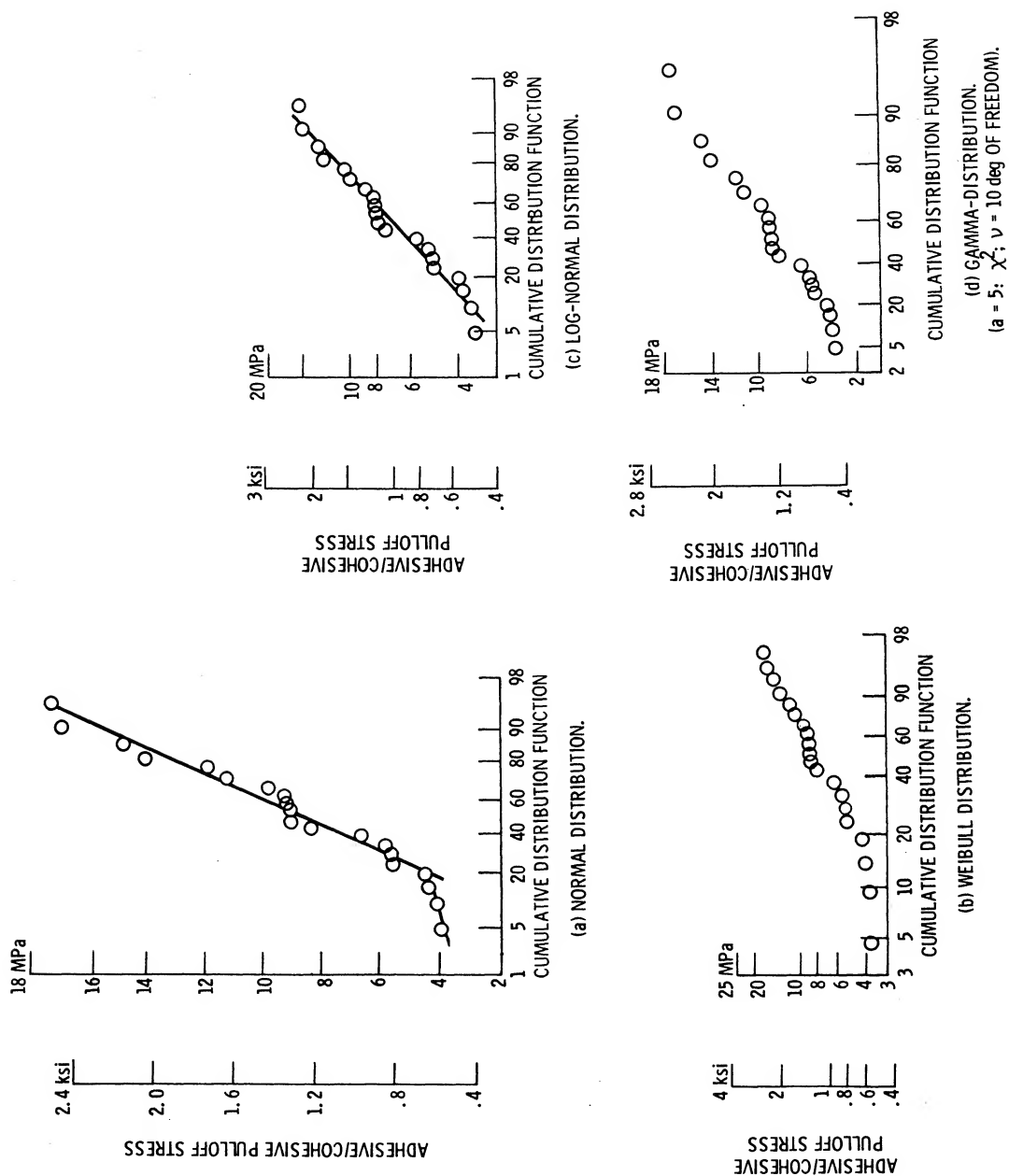


Figure 2. - Failure distribution of 20-25 mm diameter flat head thermal barrier coated adhesive/cohesive pulloff specimens.

1. Report No. NASA TM-81743		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle ASSESSMENT OF VARIATIONS IN THERMAL CYCLE LIFE DATA OF THERMAL BARRIER COATED RODS				5. Report Date	
				6. Performing Organization Code 506-53-12	
7. Author(s) R. C. Hendricks and G. McDonald				8. Performing Organization Report No. E-796	
				10. Work Unit No.	
9. Performing Organization Name and Address National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135				11. Contract or Grant No.	
				13. Type of Report and Period Covered Technical Memorandum	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes Prepared for the International Conference on Metallurgical Coatings sponsored by the American Vacuum Society, San Francisco, California, April 6-10, 1981.					
16. Abstract An analysis of thermal cycle life data for 22 thermal barrier coated (TBC) specimens is presented. The $ZrO_2-8Y_2O_3/NiCrAlY$ plasma spray coated Rene 41 rods were tested in a Mach 0.3-Jet A/air burner flame. All specimens were subjected to the same coating and subsequent test procedures in an effort to control three parametric groups; material properties, geometry and heat flux. Statistically, the data sample space had a mean of 1330 cycles with a standard deviation of 520 cycles. The data were described by normal or log-normal distributions, but other models could also apply; the sample size must be increased to clearly delineate a statistical failure model. The statistical methods were also applied to adhesive/cohesive strength data for 20 TBC discs of the same composition, with similar results. The sample space had a mean of 9 MPa (1.3 ksi) with a standard deviation of 4.2 MPa (0.6 ksi).					
17. Key Words (Suggested by Author(s)) Thermal barrier coatings Statistical methods Adhesion/cohesion Cycle life			18. Distribution Statement Unclassified - unlimited STAR Category 34		
19. Security Classif. (of this report) Unclassified		20. Security Classif. (of this page) Unclassified		21. No. of Pages	
				22. Price*	

* For sale by the National Technical Information Service, Springfield, Virginia 22161